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(19)

(54) THE PRODUCTION OF STARCH ε PROTEIN-CONTAINING POWDERS

(71) We, CPC INTERNATIONAL INC., a corporation organized and existing under the laws of the State of Delaware, United States of America, of International Plaza, City of Englewood Cliffs, State of New Jersey 07632, United States of America, do hereby declare the invention, for which we pray that a Patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to a process for obtaining water-soluble powders which simultaneously contain starch hydrolysis products and proteins which are water-soluble or have been rendered water-soluble by hydrolysis. The invention also relates to the powders resulting from these processes, and their applications.

At the present time, water-soluble starch powders or starch hydrolysis products exist which find diverse applications, especially in nutrition and in particular in the nutrition of livestock. Powders based on proteins which are suitable for the same applications are also known.

However it is known that the soluble proteins or the protein hydrolysis products are difficult to convert into powders by spraying. In effect, in accordance with the sources of these proteins, they form hygroscopic powders which rapidly absorb atmospheric moisture; these powders agglomerate into lumps and become sticky and difficult to transport.

The constantly increasing need to have available water-soluble powders which simultaneously contain starch derivatives and proteins has led the applicant company to investigate a process which simply and economically makes it possible to obtain such powders by simple spraying whilst avoiding the disadvantages inherent in the powders based on water-soluble proteins.

It is known that in order to hydrolyse and solubilise starches and proteins it suffices to subject these products to a chemical or enzymatic process or to a combination of these processes.

The process of hydrolysis and enzymatic solubilisation is the currently preferred process.

The process of enzymatic hydrolysis of natural starch is generally used in an aqueous medium. Equally the enzymatic hydrolysis of vegetable or animal proteins by means of proteolytic enzymes or proteases is carried out in an aqueous medium.

The present invention provides a process for producing starch and protein containing powders which process comprises spray-drying to a powder an aqueous composition comprising hydrolysed starch and soluble and/or hydrolysed insoluble protein made by hydrolysing an aqueous composition comprising starch and soluble and/or insoluble protein or by hydrolysing starch and insoluble protein separately and combining the hydrolysates.

Diverse sources of starch and of protein are possible. Thus there may be mentioned corn starch, wheat starch, potato starch, tapioca starch, cassava starch, rye starch, sago starch, sorghum grain starch, waxy starches, or any other natural starch. As well as the basic products, starches which have been somewhat modified by acid, alkaline or enzymatic treatments with oxidising agents, soluble or partially soluble modified starches, dextrins, pre-gelatinised products as well as any derivatives of starch and their mixtures, can be used.

As sources of proteins there may be mentioned milk serums, which may or may not be acid, demineralised or freed of lactose, residual protein—containing liquors such as corn steep liquors, the residual liquors from potato starch manufacture, cereals, cereal flours, enriched flours, basic flours, fishmeals, meat powders, dried blood, yeasts, products very rich in proteins such as corn gluten, wheat gluten, soya proteins, mycelium proteins, milk proteins.

According to a first embodiment and in accordance with the invention, a starch is

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hydrolysed in an aqueous medium containing a soluble protein. To do so, this medium is subjected to the action of an enzyme of the alpha-amylase type. When the hydrolysis has ended, this medium is concentrated, evaporated, dried and converted to a powder by any process which is in itself known.

According to a second embodiment, the medium which simultaneously contains starch and insoluble proteins is subjected to two hydrolyses: one hydrolysis for the starch, using an amylolytic enzyme of the amylase type, and another hydrolysis for the proteins, using a proteolytic enzyme of the protease type; the resulting medium is concentrated and converted to a powder.

Finally, according to a third advantageous embodiment, the mixtures resulting from the processes of the separate hydrolyses of starch and of proteins are concentrated and converted to a powder.

The appropriate techniques for powdering advantageously involve evaporation in a so-called "multiple effect" apparatus, this process being followed by the process of spray-drying.

The process of the present invention can be successfully applied to the production of non-hygroscopic protein powders starting from a medium of insoluble proteins and hydrolysing them after having taken the precaution of adding a small percentage of hydrolysed starch to this medium, or starting from an aqueous medium containing solubilised proteins and adding to this medium hydrolysed starch or starch to be hydrolysed in the latter, all these processes being carried out by a process involving concentration, drying and conversion to a powder. In effect it has been found that by following this procedure the protein particles are in some way coated with starch and are hence less hygroscopic; they do not cake together and are non-sticky and can hence easily be transported.

First embodiment: hydrolysis of the starch in a soluble protein medium.

According to this first variant, sources of soluble proteins are employed, and especially residual protein-containing liquors such as the liquors originating from the production of cheese. These residual liquors are generally very dilute. Their solids content is typically between 3% and 20%.

According to the invention, a commercial starch (starch suspension or drained starch) is added to such residual liquors up to a density of 10 to 22 Bé so as to give a proportion of starch ranging from 10 to 90% relative to the solids content of the residue. The pH is adjusted to between 5 and 8. The residual liquor is enriched in starch whilst stirring vigorously and gradually heated to 25 to 65°C, and then its temperature is raised to 80°C and finally to 99°—100°C.

The protein content is generally (depending on the type of starting material) between 2.5 and about 50% relative to the solids content. A dose of 0.001% to 0.009% of alpha-amylase is added, this dose being a function of the activity of the preparation of alpha-amylase used. The temperature is raised to 64—67°C. At that point gelatinisation takes place but after a certain time (normally after 15 minutes to 1 hour, depending on the enzyme dose used) the liquefaction is complete. The temperature is then raised to 80—88°C and the medium is kept at this temperature for the requisite time to give a degree of hydrolysis of 2 to 6 D.E. (dextrose equivalent) in the starch fraction added. The pH is then adjusted to between 4.4 and 4.8 and the temperature is raised to 100°C. The product can be passed into a homogeniser under a pressure of at least 60 kg/cm² so as to ensure more complete solubilisation of the starch.

After complete liquefaction, the product is concentrated in a double or triple effect evaporator (or in any other suitable type of evaporator) until a solids content of more than 40% is reached, and the mixture is sprayed into a drying tower equipped with a dispersing system or with any other system which allows the formation of particles. The hydrolysed starch content is between 10 and 90% and the protein content is between 10 and 50% relative to the dry material. The starch used according to this variant can originate from corn, potato or a variety of sources such as those enumerated above.

EXAMPLE 1

Corn starch and residual liquors originating from the production of cheese

These products, here described as "milk serum" can have various compositions depending on the origin and type of milk used and depending on the type of cheese produced. The product used in the present example originated from the production of cheese of the Camenbert type. It corresponds to the following analytically determined composition:

Solids content (S.C.)	15%
D.E.	53—55%, with about 75—78% lactose
Proteins	13.4%
Solubles	95.2%
Ash	10.0%

The product of 8.5 Bé strength is enriched so as to give a mixture of 50% of starch and 50% of milk serum, corresponding to a solids content of 33%.

The liquefaction or hydrolysis of the starch fraction is carried out at pH 7 by adding 0.00875% of alpha-amylase to the medium. Gelatinisation takes place at 66—67°C and liquefaction is complete at 77°C.

The incubation time required at 80—85°C to give a degree of hydrolysis of 3 D.E. is about 35 minutes.

The pH is now adjusted to 4.6 and the temperature is raised to 100°C in the course of 10 minutes.

The product is thereafter passed to the homogeniser at a pressure of 89 kg/cm², concentrated to 46% S.C. and spray-dried to give the following product:

characteristics:	
Solids content (S.C.)	95%
D.E.	28.5% (lactose: 39)
Proteins	6.6%
Solubles	92.5%
Ash	5.5%

EXAMPLE 1A

Proceeding as described in Example 1 and employing a variant, after having adjusted the pH to 4.6 and raised the temperature to 100°C, 25% and 35% of tallow are added and the hydrolysis product and the fatty matter are mixed for 10 minutes.

The product is then passed to the homogeniser at a pressure of 90 kg/cm² and is spray-dried so as to give the following product:

	25% by weight of fatty matter	35% by weight of fatty matter
Solids content (S.C.)	95%	96%
D.E.	21.8	19
Lactose	30%	26%
Proteins	5%	4.4%
Solubles	70%	62.6%
Ash	4.2%	3.6%
Fatty matter	23.8%	33.4%

By following this procedure it is thus possible to add up to 50% of fatty matter.

EXAMPLE 2

The corn steep liquors in the process for starch manufacture by the wet process, in order to produce pure starch, are also aqueous solutions containing soluble proteins extracted from the corn grains.

Starch liquefaction experiments in these steep liquors were also carried out successfully; depending on the proportion of starch added, products with varying protein content are obtained.

Corn steep liquor and corn starch

Depending on the process used and the type of corn treated, these steep liquors contain 40 to 50% of proteins and 20 to 30% of ash. The proportion of soluble solids is 92—98%.

Three experiments were carried out:

In one vat, a steep liquor with 7.6 S.C., containing 48.2% of proteins and 27% of ash, relative to dry matter, is enriched as described in Example 1 with starch until its strength is about 10 Bé, 15 Bé and 20 Bé and is treated exactly in accordance with the process described, at pH 5.8—6.2 and with 0.00875% of alpha-amylase sold under the trade name "Seclin".

The alpha-amylase content is calculated relative to dry starch.

Gelatinisation takes place and liquefaction is complete from 75—77°C onwards. The contact time at 80—85°C is continued until a degree of hydrolysis of 2.5 D.E. (calculated relative to the starch fraction) is achieved.

Reaction is stopped by lowering the pH to 4.5 and heating to 100°C.

The product is passed to the homogeniser:

a) at 88 kg/cm² pressure for the 10 Bé medium
b) at 96 kg/cm² pressure for the 15 Bé medium
c) at 130 kg/cm² pressure for the 20 Bé medium

and concentrated to 41.3% in the case of a), to 42.6% in the case of b) and to 40.6% in the case of c).

The products are thereafter sprayed and the products resulting from this operation give the following analysis:

	a)	b)	c)
Proteins	30%	18%	10%
S.C.	94.3%	95.2%	98.4%
pH	6.0	6.0	4.7
Ash	15.5%	11.1%	8.0%
Solubles	90.0%	86.0%	80.0%
D.E. (originating from the malto-dextrins)	1.7%	0.6%	0.4%

EXAMPLE 3

In the manufacture of potato starch, the potato wash liquors, which are rich in proteins, can also serve for the suspension and hydrolysis of the starch.

The method of working is the same as that described above.

In the experiments carried out, the residual liquor from potato starch manufacture had the following composition:

Solids content	7%
Proteins	42%
Ash	17% (relative to solids content)

This liquor, which is rich in proteins, is enriched with suspended corn starch. Varying amounts of starch can be added; in the present example the composition of the mixture was:

Solids content (S.C.)	21%
Proteins	14%
Starch	66%
Ash	6%

This suspension of starch in the protein-containing water from potato starch manufacture is now adjusted to pH: 6.5, the calcium content is adjusted to 350 ppm, and 0.015% of alpha-amplase is added.

The mass is rapidly heated to 85°C and kept at this temperature for 40 minutes. Thereafter it is heated to the boil and kept thereat for 5 minutes so as to destroy the enzymatic activity.

This solution of liquefied starch and of proteins is now concentrated to 55% S.C. by evaporation in vacuo.

After spray-drying, a powder of the following composition is obtained:

Moisture content	6.3%
Solubles	95.4%
D.E.	2.8%
Proteins	14.3%
Ash	6.8%

This powder, composed of proteins and starch hydrolysis products, thus contains malto-dextrins and can be used in livestock nutrition.

All the products obtained in the course of the experiments carried out are easier to spray-dry, less hygroscopic and more mobile than the residual liquors spray-dried as such. More particularly, it proved possible for the first time to spray the residual liquors from the manufacture of monosodium glutamate.

As residual liquors rich in proteins there may also be mentioned the liquors from fisheries and fish canneries, the broths from the culture of antibiotics.

Second embodiment

To obtain powder products containing hydrolysis products of starch and proteins, the starting materials must be products which simultaneously contain starch and proteins, such as: cereals, cereal flours, enriched flours, basic flours.

According to this embodiment of the invention, the starch is first treated with alpha-amylase under the conditions described in the above examples.

When the starch part has been hydrolysed and dextrinised, the enzymatic action being destroyed, the proteins are next hydrolysed, this being done by the action of a proteolytic enzyme of the protease type under the conditions of the examples which follow. The mixture of the two hydrolysis products, namely of starch and of proteins, is now concentrated in vacuo and converted to a powder, preferably by spraying.

EXAMPLE 4

Normal flour

A wheat flour (50 kg) is suspended in water.

Characteristics of the suspension:

Solids content (S.C.)	31%
Starch	80%
Proteins	11%

This suspension is adjusted to pH 6.5 and 300 ppm of calcium are added thereto.

The alpha-amylase is added to the suspension at the rate of 0.012% relative to dry starch.

The temperature of the suspension is now raised to 85°C and maintained for 60 minutes. After this incubation time, the starch is dextrinised and liquefied and the whole is heated to the boil and kept thereat for 10 minutes.

This mass is thereafter cooled to 60°C, the pH is raised to about 8.2 by means of sodium carbonate and a new type of enzyme is added; this is a proteolytic enzyme of the group of the proteases. In the present case, "Alcalase" (Registered Trade Mark) of Messrs. NOVO —Copenhagen (Denmark) was used. The dose of this enzyme is 0.05% relative to total solids. After 10 hours incubation, during which the pH is constantly maintained by regular addition of Na_2CO_3, the temperature is raised to about 100°C to destroy the proteolytic activity.

It is found that the protein has become very soluble.

This liquid containing the starch hydrolysis product and the protein hydrolysis product is now concentrated to 56% solids content and dried, and then converted to a powder by spraying.

EXAMPLE 5

Flour enriched in gluten

A wheat flour, suspended in water, was beforehand treated by centrifuging in a super-decanter of the "Alpha-Laval" type.

The products obtained are, firstly, a depleted flour containing about 2 to 3% of proteins and, secondly, a suspension having the following characteristics:

Solids content (S.C.)	18%
Proteins	34%
Starch	61%

This suspension is treated as above.

The proteolytic enzyme dose is 12%.

On using the technique described above, a powder which simultaneously contains a starch hydrolysis product and a protein hydrolysis product (soluble protein, polypeptide and amino-acids) is obtained.

Composition of the powder obtained

Moisture content	6.8%
Soluble matter	94.0%
Soluble proteins	33.6%
D.E.	3.1%
Ash	3.2%

EXAMPLE 6

Starting from grits of corn which has been degerminated dry, or starting from corn which has beforehand been steeped and degerminated wet, an amylaceous product containing about 10 to 12% of protein is obtained.

In the case of this starting material, the starch is hydrolysed by the enzymatic method described above.

A mixture of starch hydrolysis product and insoluble proteins is thus obtained.

At this stage, the technique of proteolytic hydrolysis by means of protease is applied, under the known conditions. The mixture is then filtered to remove the fibrous and cellulosic residue. The solution is then concentrated, dried and converted to a powder by spraying.

The grits and flour part of the corn which was degerminated dry is now suspended in water; the pH is adjusted to 6.5 and the alpha-amylase enzyme is added to give a content of 0.015% relative to S.C.

The mixture of water, corn grits and flour is then heated to 85°C and kept thereat.

Characteristics of the suspension

Solids content (S.C.)	30%
Proteins	10.5%
Starch	86.1%
Fats	1.6%
Cellulose	0.43%

After treatment with alpha-amylase, a product of the following composition is obtained:

Solids content (S.C.)	32%	
Proteins	10.5%	
D.E.	4.2%	relative to solids content
Soluble matter	84.8%	
Fats	1.6%	
Cellulose	0.43%	

This product is now cooled and subjected to the action of a proteolytic enzyme for 15 hours at a temperature of 60°C and at pH 8. The dose of the protease (Alcalase Novo) is 0.04% relative to solids content.

After the incubation period, the pH is adjusted to 5 and the resulting mass is then sieved on a press, or by any other process for solids-liquid separation.

The residue from this separation is dried and can be used in animal nutrition.

Before sieving, the combined material contained, in addition to fats and celluloses:

Solids content	32%
Soluble matter	93%
Soluble proteins	8.3%
Insoluble proteins	2.1%

After sieving on a filter press and washing the residue, which was used in these tests, the soluble matter had the following composition:

Solids content	21%
Soluble proteins	9.2%
Malto-dextrin	89%
Ash	1.8%

The filtration residue corresponds to the following composition:

Proteins	about 24% (a part of the proteins not being hydrolysed)
Malto-dextrin	about 22%
Fats	about 15.4%
Cellulose	about 4.8%

The soluble components obtained by this treatment are now concentrated by evaporation in vacuo and converted to a powder by spray-drying.

The product obtained has the following characteristics:

Moisture content	6.5%
Proteins	9.2%
Malto-dextrin of 7 D.E.	89%
Ash	1.8%
D.E.	6.3%

In the manufacture of corn starch by the wet method, there exists, at various stages of the purification, starch-protein systems which

are progressively richer in proteins, namely from 20 to 70%. These substrates can thus be used as a suitable starting material in the process of the invention and make it possible to obtain hydrolysed starch and protein products in powder form which contain varying amounts of soluble proteins.

Any other product containing proteins and starch can be treated in the same manner.

Third embodiment

This third embodiment consists of hydrolysing products which are very rich in proteins, such as corn gluten, wheat gluten, yeasts, soya proteins, fish proteins, mycelium proteins, milk proteins.

These proteins are hydrolysed by proteolytic enzymes. These hydrolysis products are then mixed with the previously concentrated hydrolysis products of pure starch after which the mixture is spray-dried.

EXAMPLE 7

The product is treated by the same enzymatic methods as those described above.

The product originating from the manufacture of corn starch by the wet method is a suspension of starch and proteins. Its composition, relative to solids content, is the following:

Proteins	60%
Starch	30%
Fats	3.8%
Ash	1.2%

The suspension had a concentration of 30%.

The starch fraction is first liquefied by means of alpha-amylase and thereafter the proteins are hydrolysed enzymatically. If the whole is concentrated in vacuo and converted to a powder by spray-drying, as above, the final product shows the following characteristics:

Moisture content	5.2%
Soluble proteins	59.1%
D.E.	1.8%
Soluble matter	93%
Fats	3.8%

Now the above protein and starch hydrolysis products can also be mixed with a hydrolysis product of pure starch having a D.E. of 5, so as to adjust the protein content to about 30%.

This mixture of two hydrolysis products, firstly starch-proteins and secondly starch, is concentrated and again converted to a powder. The composition of the product obtained is the following:

Moisture content	5.8%
Soluble proteins	28.3%
Total proteins	30.8%
D.E.	1.1%
Soluble matter	92.3%
Fats	1.8%

EXAMPLE 8

A fish protein in the form of a powder is suspended in water in the presence of a proteolytic enzyme which is specific for animal proteins, sold under the trade name "Newlase" by Messrs. Amano Cy. of Japan.

The working conditions are: pH 5.3; temperature 50°C.

After 8 hours' incubation, the protein hydrolysis product is enriched with liquid malto-dextrins to 5 D.E.

The whole is then evaporated and converted into a powder.

Instead of adding previously prepared starch hydrolysis products to the hydrolysed protein, it is possible to incorporate or suspend starch in the protein hydrolysate and then subject it to an α-amylase hydrolysis in a way analagous to that described in the first embodiment.

WHAT WE CLAIM IS:—

1. A process for producing starch and protein containing powders which process comprises spray-drying to a powder an aqueous composition comprising hydrolysed starch and soluble and/or hydrolysed insoluble protein made by hydrolysing an aqueous composition comprising starch and soluble and/or insoluble protein or by hydrolysing starch and insoluble protein separately and combining the hydrolysates.

2. A process as claimed in claim 1 wherein the hydrolysis of the starch is carried out in an aqueous medium containing a soluble protein and that this medium is subjected to the action of enzymes of the alpha-amylase type.

3. A process as claimed in claim 1, wherein the medium which contains both starch and insoluble proteins is subjected to two hydrolyses, one to hydrolyse the starch and the other to hydrolyse the proteins, under the action of two specific enzymes, and that the resulting medium is concentrated and spray-dried into a powder.

4. A process as claimed in any one of claims 1 to 3, wherein the medium to be spray-dried furthermore contains a fatty material.

5. A process as claimed in any one of claims 1 to 4, wherein the starch originates from corn, potato, cassava, wheat or rye.

6. A process as claimed in any of claims 1 to 5 wherein the protein is of vegetable origin from cereals, oleaginous products, wheat, soya, beet or yeasts or of animal origin from casein, fish meal, meat powder or dried blood.

7. A process as claimed in any of claims 1 to 6, and substantially as herein described in any one of Examples 1 to 8.

8. Powder based on starch and on proteins, obtained by the process according to any one of claims 1 to 7.

9. A powder as claimed in claim 8, wherein the proportion of starch is between 2% and 98% and the proportion of proteins is between 98 and 2%.

10. A powder as claimed in either claim 8 or claim 9, wherein furthermore contains up to 50% by weight of a fatty material relative to the total weight of the powder.

11. A foodstuff comprising the powder claimed in any one of claims 8 to 12.

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